STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

QUARTERLY PROGRESS REPORT 5

Covering the Period

June 26, 1965 to September 26, 1965

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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I. INTRODUCTION

The program reviewed in this fifth quarterly progress report, covering the period from June 26, 1965 to September 26, 1965, is sponsored by the National Aeronautics and Space Administration. Its purpose it to examine the influence of stress on the corrosion behavior of an advanced refractory alloy in potassium (Task I) and to investigate corrosion mass transfer effects in a stainless steel - columbium alloy - potassium system (Task II).

Task I

While there is considerable evidence that refractory alloys have excellent corrosion resistance to potassium, there are few experiments which describe the possible effects of stress on corrosion when the stress is sufficiently large to produce substantial amounts of creep during the test. It is appropriate for comparative purposes to study an advanced refractory alloy which has demonstrated excellent corrosion resistance to refluxing potassium during long-time exposures conducted at relatively low stresses at 2000°F. In this regard, D-43 columbium base alloy, in the form of welded capsules, has been tested in potassium under refluxing conditions for periods of 5000 and 10,000 hours at temperatures on the order of 2000°F (1) and has been selected for inclusion in this program.

The D-43 alloy reflux capsules shall be tested under conditions which result in about 5 to 10% strain during a 500- to 2000-hour exposure period in the 2000° to 2200°F temperature range. The reflux capsules used in this study will be of similar size to those previously described (1). The capsule wall shall be reduced in the potassium liquid region and in the vapor condensing region to provide gauge sections where the extent of creep can be measured. Moderate temperature adjustment can be made during the test to achieve the desired strain-time conditions.

Task II

The use of stainless steel, rather than refractory alloys, for power plant radiator construction and for the lower temperature portion of experimental facilities constitutes material and fabrication cost savings. Two methods of employing this approach are: use of co-extruded, stainless steel shell-refractory alloy core, tubing in the radiator or use of an all stainless steel radiator joined to the system by a bimetallic joint. Although the latter approach is preferred considering cost and problems associated with fabrication and joining of co-extruded tubing, a major uncertainty and limitation arises from the mass transfer of interstitial elements from the stainless steel to the refractory alloys through the alkali metal.

It is well established that the carbon and nitrogen transfer from Type 316SS to Cb-1Zr alloy at temperatures near 1500°F (2). While some important aspects of this mass transfer behavior have been examined, several critical details require additional investigation. There is a need to define acceptable time and temperature conditions of operation in terms of maintaining satisfactory performance of the refractory alloys, such as Cb-1Zr alloy. Also, there are certain metallurgical aspects of this behavior which should be investigated in an effort to eliminate or reduce the mass transfer rate. In the latter category, it is most appropriate to consider the stabilization of carbon and nitrogen in the stainless steel by the addition of metallic elements which form carbides and nitrides of high thermodynamic stability. Commercially available, titanium stabilized, Type 321SS is one such alloy. A comparative investigation of this alloy and Type 316SS was performed to indicate the ability of the titanium addition to reduce or eliminate interstitial mass transfer in a stainless steel Cb-1Zr alloy bimetallic system. Columbium-1% zirconium alloy specimens were exposed to liquid potassium in Type 321SS and Type 316SS capsules for 1000 hours at 1400°F under isothermal conditions to evaluate this premise. Post-test evaluation showed that Type 321SS has a significant advantage over Type 316SS in refractory metal-stainless steel-potassium system in inhibiting mass transfer of the interstitial elements carbon and nitrogen from the stainless steel to the refractory metal.

II. SUMMARY

During the fifth quarter of this program, the topics abstracted below were covered. The results are interpretatively presented in this report.

Task I - Stress Corrosion Reflux Capsule Tests

The D-43 Alloy Stress Corrosion Reflux Capsule Test I completed 1000 hours of testing at a nominal temperature of 2100°F and was removed from the test facility. Equivalent uniaxial creep strains of 7.4% in the liquid region and 5.4% in the condensing region were calculated from the expansion data. The capsule was drained of potassium, cleaned and sectioned. Although examination of the inner surface of the capsule revealed the presence of spots in the liquid and condensing regions, further investigations indicated no association between the location of the spots and the regions of high stress. Lower uniaxial creep strengths were obtained from capsule test data than were predicted by pretest and literature data. Additional uniaxial creep tests are planned in expectation of explaining this anomally.

The D-43 Alloy Stress Corrosion Reflux Capsule Test II continues on test at a nominal temperature of 2050°F. A total of 1600 hours of test time has been accumulated.

Task II - Bimetallic Isothermal Capsule Tests

A Topical Report has been written on Task II and will be submitted to the NASA Technical Manager for review during the next reporting period.

III. TASK I - STRESS CORROSION REFLUX CAPSULE TESTS

D-43 Alloy Stress Corrosion Reflux Capsule Test I

Capsule Test I completed 1000 hours of exposure at a nominal temperature of 2100°F without difficulty. Subsequently, the capsule was removed from the test facility and examined, Figure 1. Lüders lines were apparent on the expanded liquid and condensing regions as previously evidenced in the preliminary capsule test. The diametral expansion was checked with a micrometer and proved to be in excellent agreement with that indicated in the LVDT measurements. As an example, the 0.0896-inch diametral change measured in the liquid region by the LVDT's, measured 0.088-inch with the micrometer. This represents an LVDT accuracy of better than ± 2%. Total equivalent uniaxial creep strains of 7.4% in the liquid region and 5.4% in the condensing region were calculated from the diametral change. The creep curves plotted from the LVDT data are depicted in Figure 2. The lower creep strain measured in the condensing region results partially from the 15°-20°F lower temperature in the condensing region over that which was measured in the liquid region. The pressure-time data for Capsule Test I is shown in Figure 3.

The capsule was opened and drained of potassium in an argon atmosphere. Subsequently, the capsule was cleaned by vacuum distillation at a temperature of about 600°F and a pressure of 5×10^{-5} torr and was sectioned, Figure 4. Spots were observed on the ID surface of the capsule and are shown at higher magnification in Figure 5. The fact that these spots appear in the thick-wall section of the capsule as well as in the reduced-wall section suggests their presence is not dependent upon the existance of high stress. Similar spots

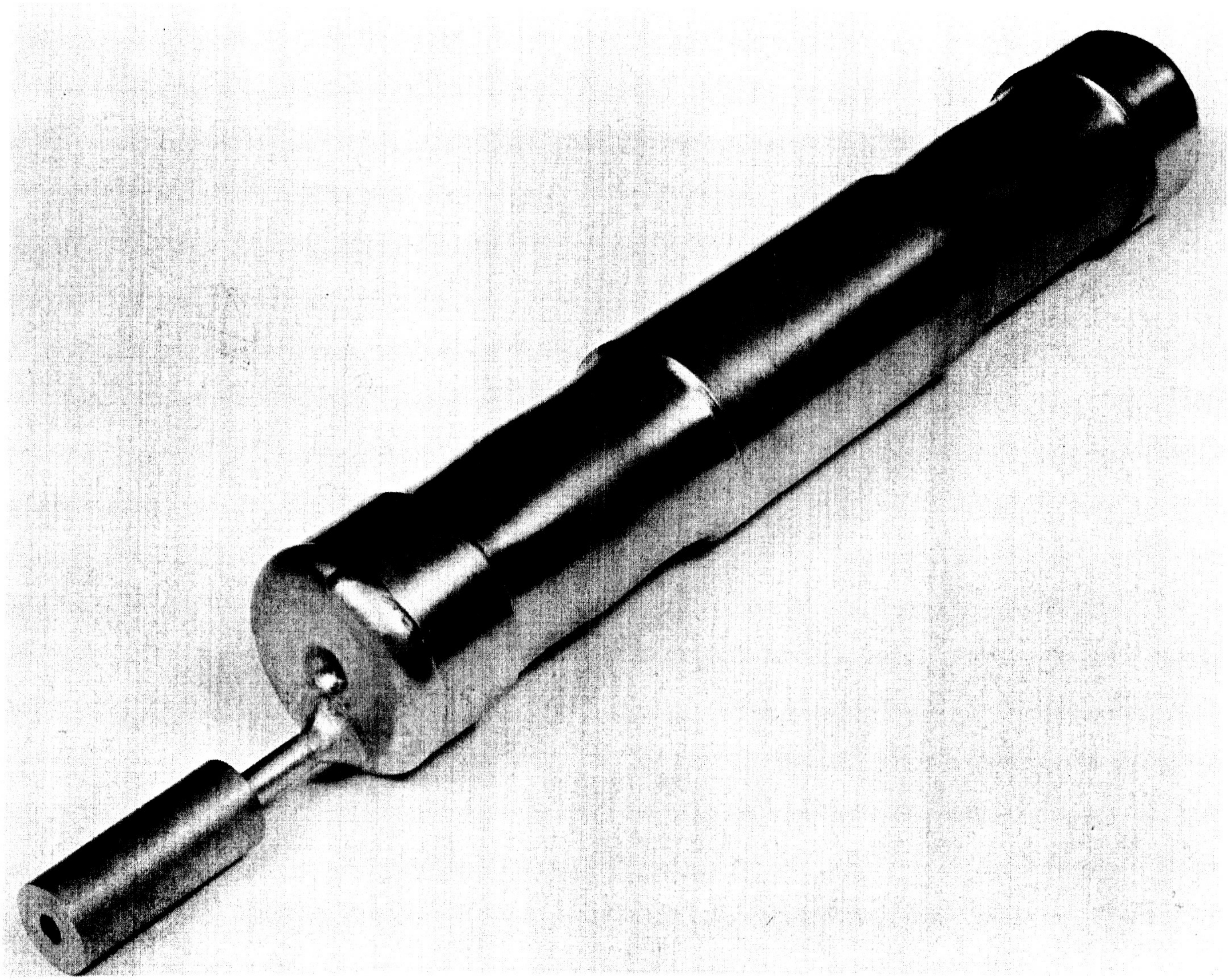

Figure 1. D-43 Alloy Stress Corrosion Reflux Capsule I After 1,000 Hours of Testing at 2100°F in a Vacuum of 1.2×10^{-8} Torr and Prior to Removal of the Contained Potassium.

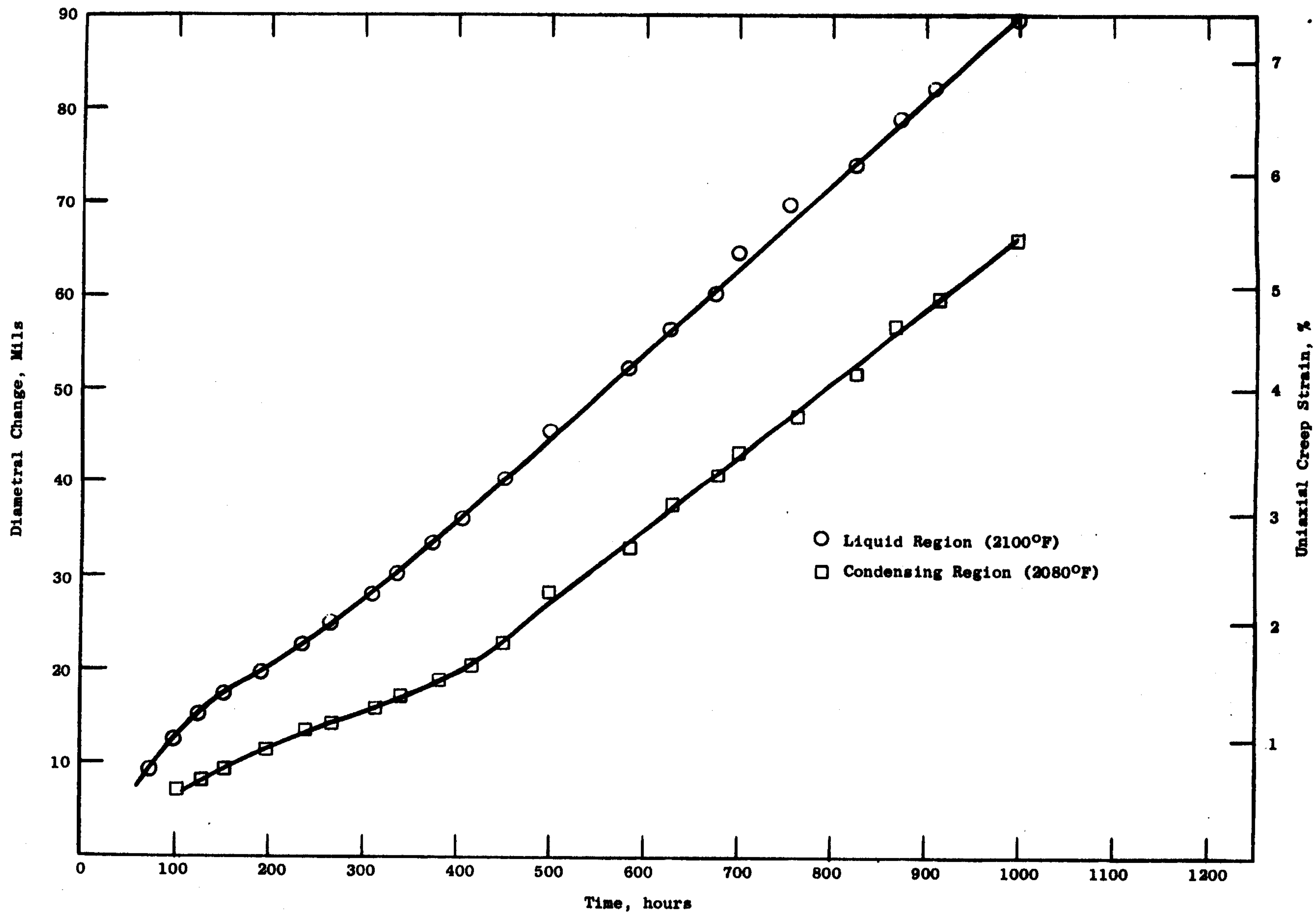

Figure 2. Biaxial Creep Data on the D-43 Alloy Stress Corrosion Reflux Capsule Test I - 1,000 Hours Exposure to Potassium.
Calculated Uniaxial Stress-6100 psi.

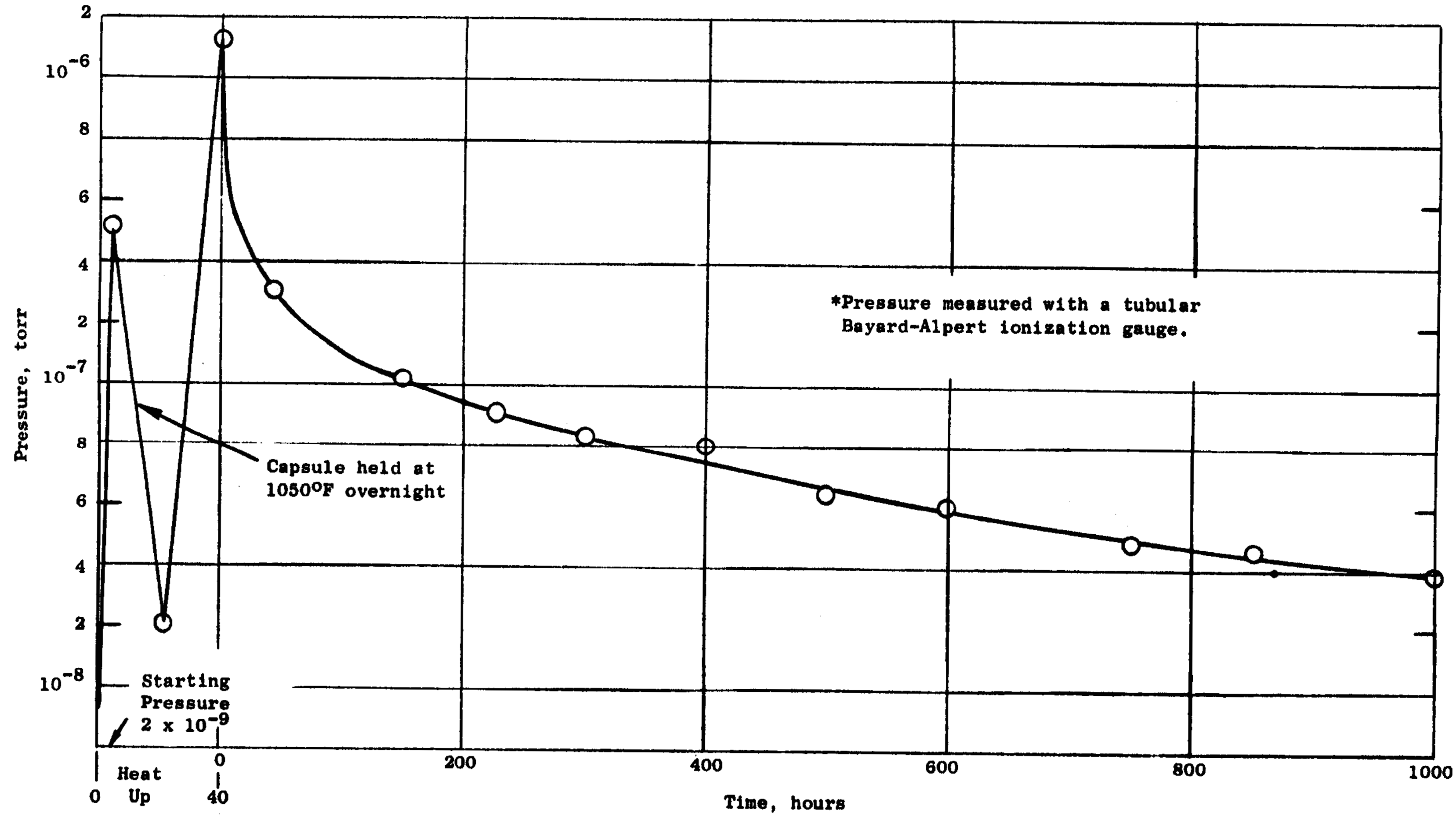

Figure 3. Pressure-Time Measurements for the D-43 Alloy Stress Corrosion Reflux Capsule Test I.*

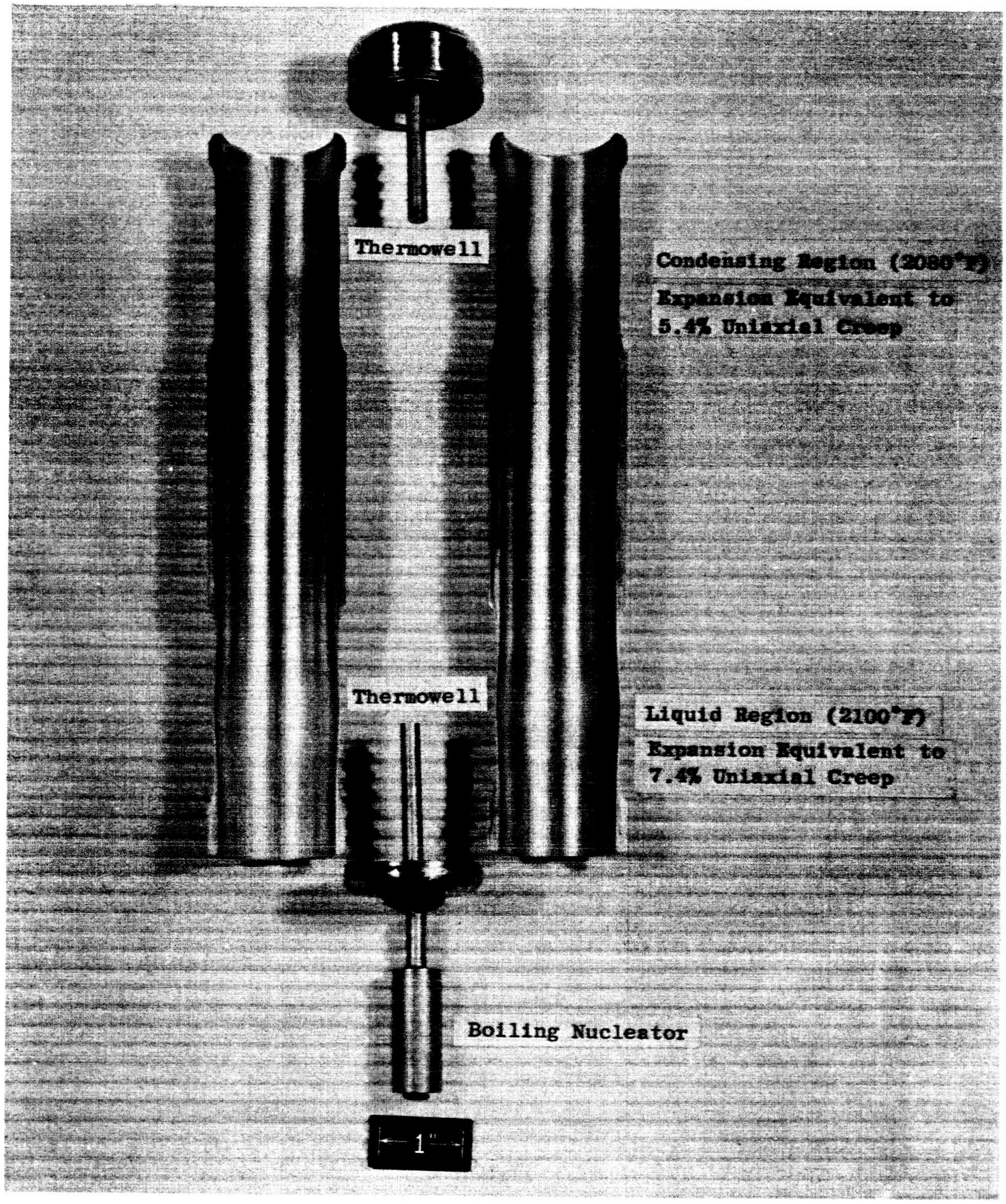

Figure 4. Sectioned D-43 Alloy Stress Corrosion Reflux Capsule I After a 1,000-Hour Exposure to Potassium at 2100°F.

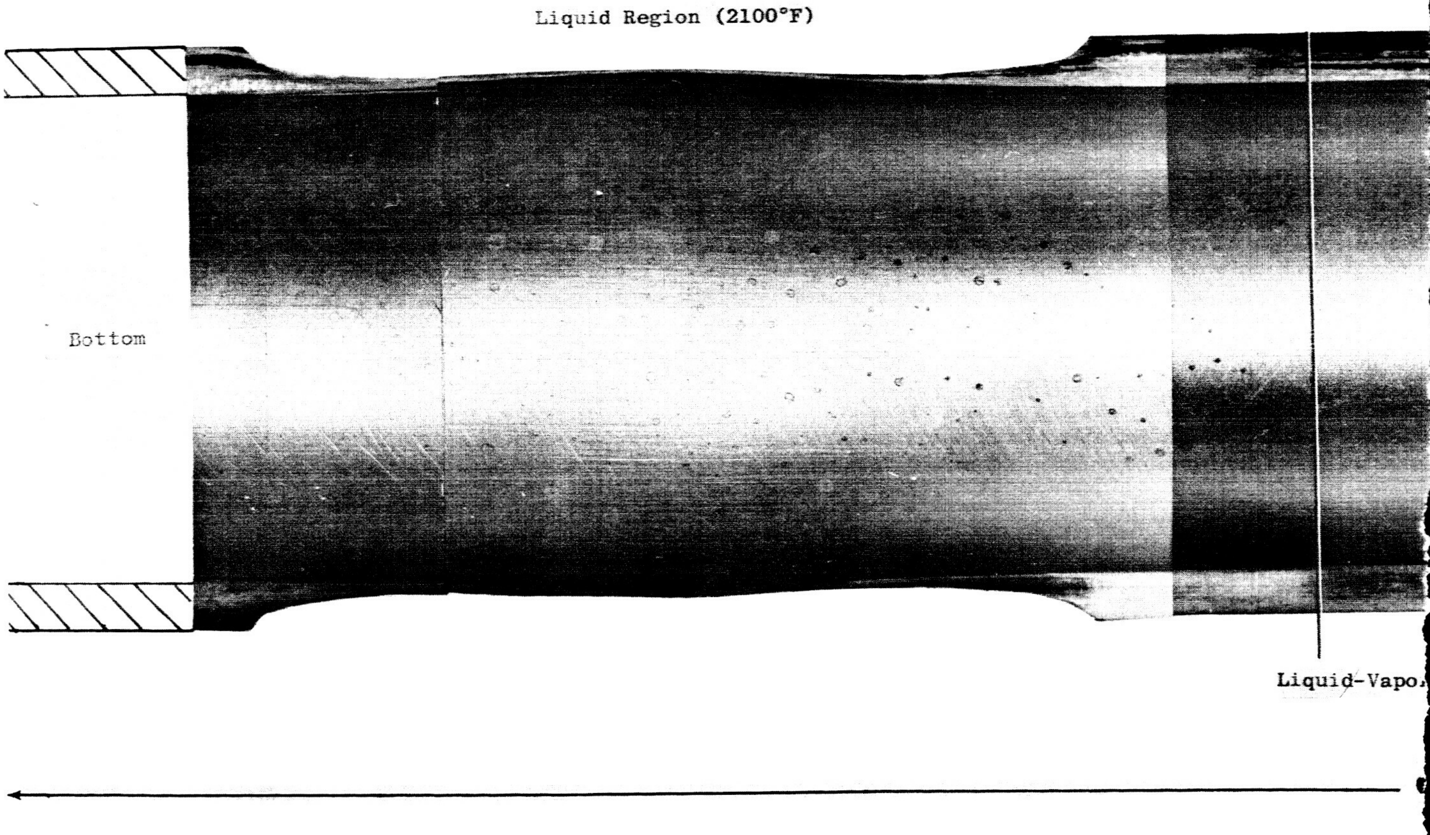

Figure 5. The ID Surface of D-43 Alloy
1000-Hour Exposure to Potassi
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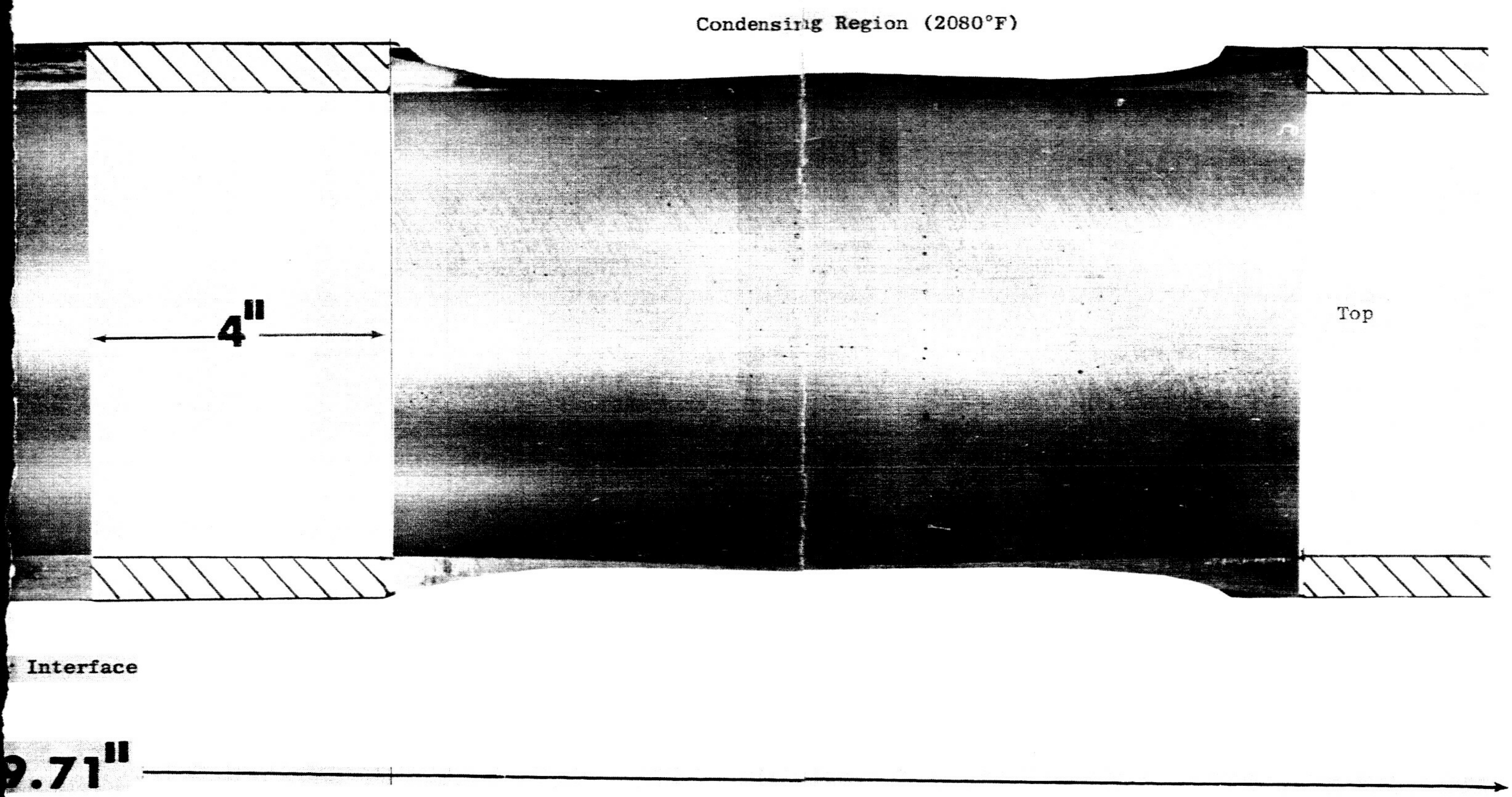

Stress Corrosion Reflux Capsule I After a
um at 2100°F. Spots can be Observed in
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2 3

were observed in the condensing region of the preliminary capsule test (3). However post-test evaluation of D-43 alloy capsule tested previously for 5000 and 10,000 hours did not delineate spots. Therefore, in an effort to explain this anomally, a study of the capsule preparation and cleaning procedures employed in recent capsule tests at General Electric was made. The results of this study are shown in Table I.

The 5000 and 10,000-hour D-43 capsules were post-test cleaned by draining and reacting the residual potassium with ethanol in hexane. This procedure was found to be less desirable than vacuum distillation as the ethanol reaction with potassium can result in hydrogen pickup in the refractory alloys (4). As a result, succeeding capsules, including those in this study, were cleaned by vacuum distillation. No spots were observed in the Cb-1Zr capsules containing Mo-TZM insert specimens which were cleaned by vacuum distillation without prior draining (5). In order to remove this one variable, the D-43 Stress Corrosion Reflux Capsule Test II presently on test, will be cleaned by the ethanol-hexane technique*. Any hydrogen pickup resulting from this cleaning procedure will not jeopardize the post-test evaluation of this capsule as mechanical property tests are not planned.

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*On October 14, 1965, D-43 Alloy Reflux Capsule Test II completed 2000 hours of testing at 2050°F. The capsule was removed from the test facility and placed in the E.B. Welding Chamber. Subsequent to evacuating the chamber to 1×10^{-5} torr and back filling with bottle argon containing less than 3 ppm oxygen, the capsule was opened, drained of potassium, and cleaned by reacting the residual potassium with ethanol in hexane. Subsequently, the capsule was sectioned and examined. No spots were noted on the capsule ID. This finding suggests that the presence of spots in the previous capsules tested in this program was a function of cleaning procedure and not due to potassium exposure in testing. The mechanism by which these spots formed during vacuum distillation after draining the capsule is not completely understood at this time.

TABLE I. CAPSULE PREPARATION AND CLEANING PROCEDURES

Capsule Test	Raw Material	Fabrication	Pretest Cleaning	Post-Weld Heat Treatment	Post-Test Cleaning	Observations
I. D-43 Alloy Stress Corrosion Capsules						
A. Preliminary Test	Bar Recrystallized 1 Hour @ 2600°F	Machined 16 rms Finish on ID	Vapor Degreased* Ethanol Wash Air Dry	None	Drain & Vacuum Distillation 600°F & 5×10^{-4} Torr	Spots on ID
B. Capsule Test I	"	"	"	1 Hour @ 2400°F 10^{-6} Torr, Double Wrapped in Cb-1Zr Foil	"	"
C. Capsule Test II	"	"	"	"	On Test	---
II. D-43 Alloy Capsule Tests[1] (5000 & 10,000 Hours)	Sheet Stress-Relieved 1 Hour @ 2200°F	Rolled & Welded Sheet	Pickle HNO_3-HF-H_2O, Water Rinse, Ethanol Wash, Air Dry	1 Hour @ 2400°F 10^{-5} Torr, Not Wrapped	Drain & React with Hexane & Ethanol	No Spots Observed
III. Mo-TZM-Cb-1Zr Capsule[5] (2500 & 5000 Hours)	Cb-1Zr Tubing	Gun Drilled & Honed to 32 rms	Enthanol Wash, Air Dry	None	Vacuum Distillation Without Draining 800°F x 5×10^{-4} Torr	No Spots Observed

* Thermowells and Caps were not Vapor Degreased but Cleaned in Ethanol and Dried. Spots were Observed on these Pieces after Post-Test Cleaning.

As described previously (3), the uniaxial creep data obtained from pretest evaluation of the D-43 alloy used for capsule fabrication indicated a higher strength than that which was obtained in the actual capsule test. The capsule test data also indicated lower creep strengths than that reported in the literature (6). A graphical comparison of these data is shown in Figure 6. Pretest creep data were obtained with a 0.160-inch diameter gauge section on bulk type specimens in a facility capable of 10^{-6} torr vacuum (7). An additional D-43 bulk specimen has been submitted for creep testing in a facility capable of 10^{-10} torr vacuum, Figure 7. Results of this test should indicate if the higher pretest data were a function of test environment although post-test chemical analyses of those creep specimens indicated negligible contamination (7). The effects of specimen geometry are also being investigated since the DMIC data were obtained on sheet specimens (6) and the capsule data were indicative of creep in a 0.020-inch thick wall section. As a result, 0.020-inch thick creep specimens have been machined from the 0.140-inch wall section of the D-43 alloy preliminary capsule for creep testing at 2100°F in a 10^{-8} torr vacuum. The short time this capsule material was exposed at temperature (3) should not influence its metallurgical structure and the resulting strengths should be representative of the recrystallized D-43 alloy used to fabricate Capsule I.

D-43 Alloy Stress Corrosion Reflux Capsule Test II

An additional stress corrosion reflux capsule was filled with sufficient potassium to produce a 5-inch liquid height at the test temperature (2050°F). The potassium was transferred directly to the capsule from the final hot trapping container and the capsule was sealed by electron beam welding in a vacuum of 8×10^{-6} torr. The potassium that was used for filling the capsule

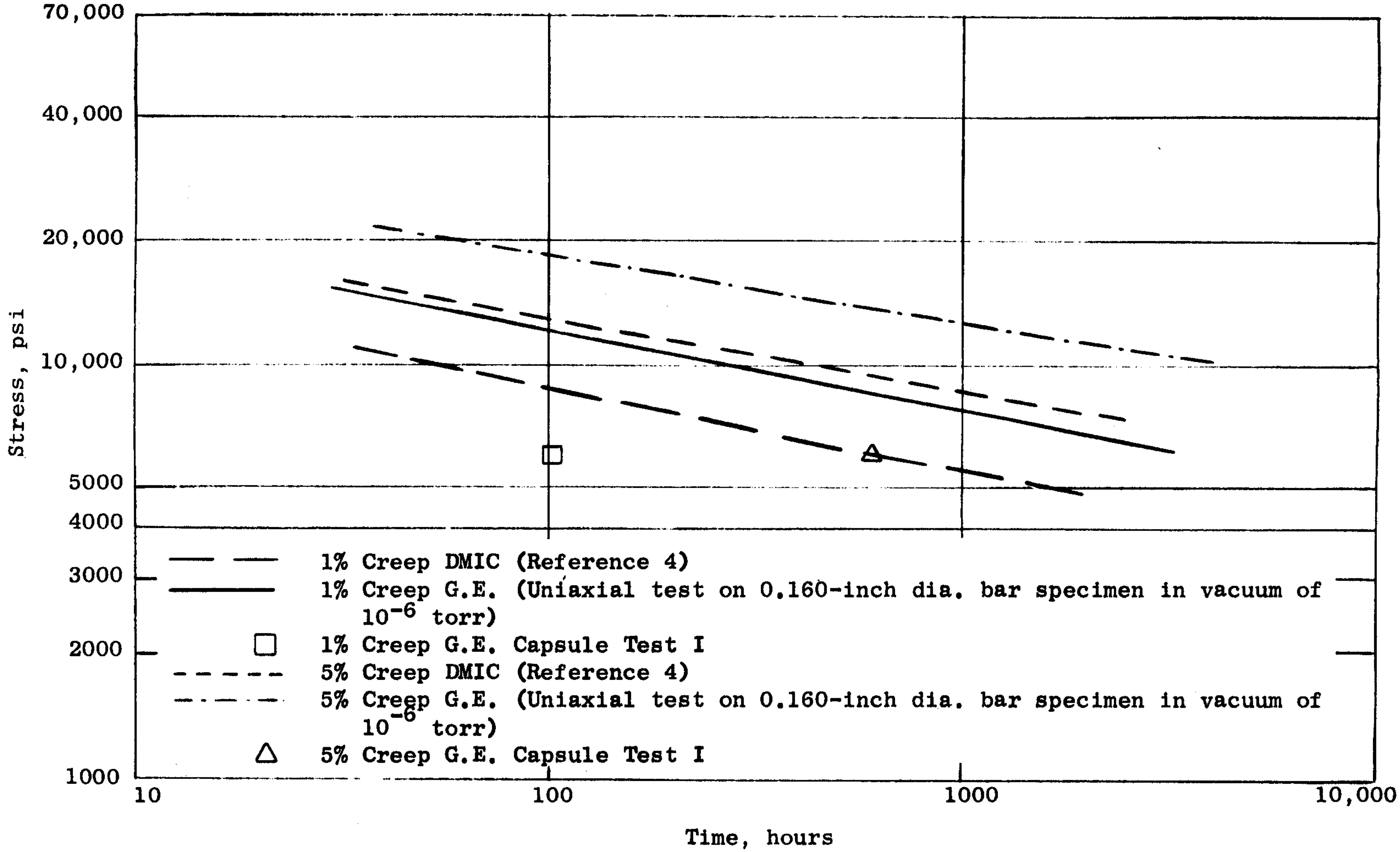

Figure 6. Preliminary Comparison of Creep Properties of D-43 Alloy at 2100°F.*

*A complete comparison of data obtained in capsule tests and uniaxial creep tests will be reported in the topical report.

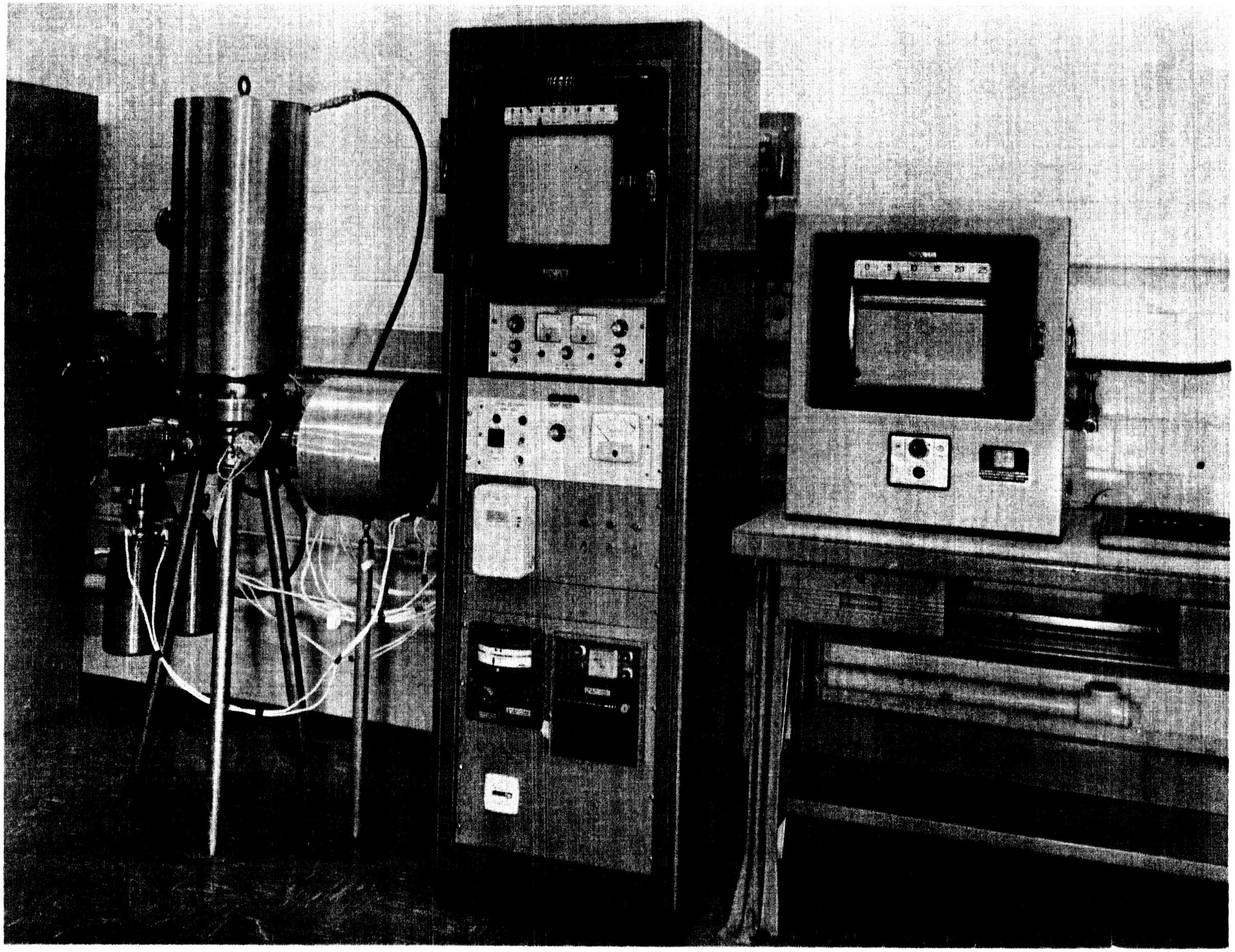

Figure 7. High-Vacuum Creep-Rupture Facility Capable of Achieving a Pressure of 10^{-10} Torr and Incorporating a 100 Liter/Sec Getter-Ion Pump.

was sampled at the same time that the capsule was filled and was analyzed for oxygen by the mercury amalgamation method (helium cover gas); the results showed the oxygen in the potassium taken from the fill tube to be 6 ppm and the oxygen in the potassium taken from the chamber to be 11 ppm. Spectrographic analyses of this potassium and that used to fill Capsule I are presented in Table II. The filled and sealed capsule was examined radiographically to assure a sound electron beam weld and subsequently was installed in the test facility.

After a thorough instrumentation check, the LVDT's were calibrated and the system was closed and evacuated. Mass spectrometer leak checking indicated no leaks present and pre-bakeout vacuum of 8×10^{-8} torr was achieved. The system was baked out at 350°C for six hours; on cooling, a vacuum of 1.5×10^{-9} torr was measured with a tubular Bayard-Alpert ionization gauge in the chamber wall. Capsule testing was initiated on 7-22-65 and at the end of this reporting period 1600 hours of test time have been accumulated at a liquid region temperature of 2050°F. A vacuum of 1.2×10^{-8} torr was recorded at that time. The creep data obtained to-date is depicted in Figure 8. As was the case in Capsule Test I, creep in the condensing region is less than that measured in the liquid region.

IV. TASK II - BIMETALLIC ISOTHERMAL CAPSULE TESTS

A Topical Report has been prepared and will be submitted for NASA review within the next reporting period.

TABLE II. SPECTROGRAPHIC ANALYSIS OF THE POTASSIUM USED TO FILL THE D-43 ALLOY STRESS CORROSION REFLUX CAPSULES

Element	Analyses[1], ppm	
	Capsule Test I	Capsule Test II
Ag	<1	<1
Al	<1	1
Be	1	<1
Ca	1	>25
Cb	<1	<5
Co	<1	<1
Cr	<1	<1
Cu	<1	<1
Fe	1	<1
Mg	<1	15
Mn	<1	<1
Mo	<1	<1
Na	1	5
Ni	<1	<1
Pb	<1	<1
Si	1	<5
Sn	<5	<5
Ti	<1	<1
V	-	<5
Zr	<5	<5

[1] As KCl

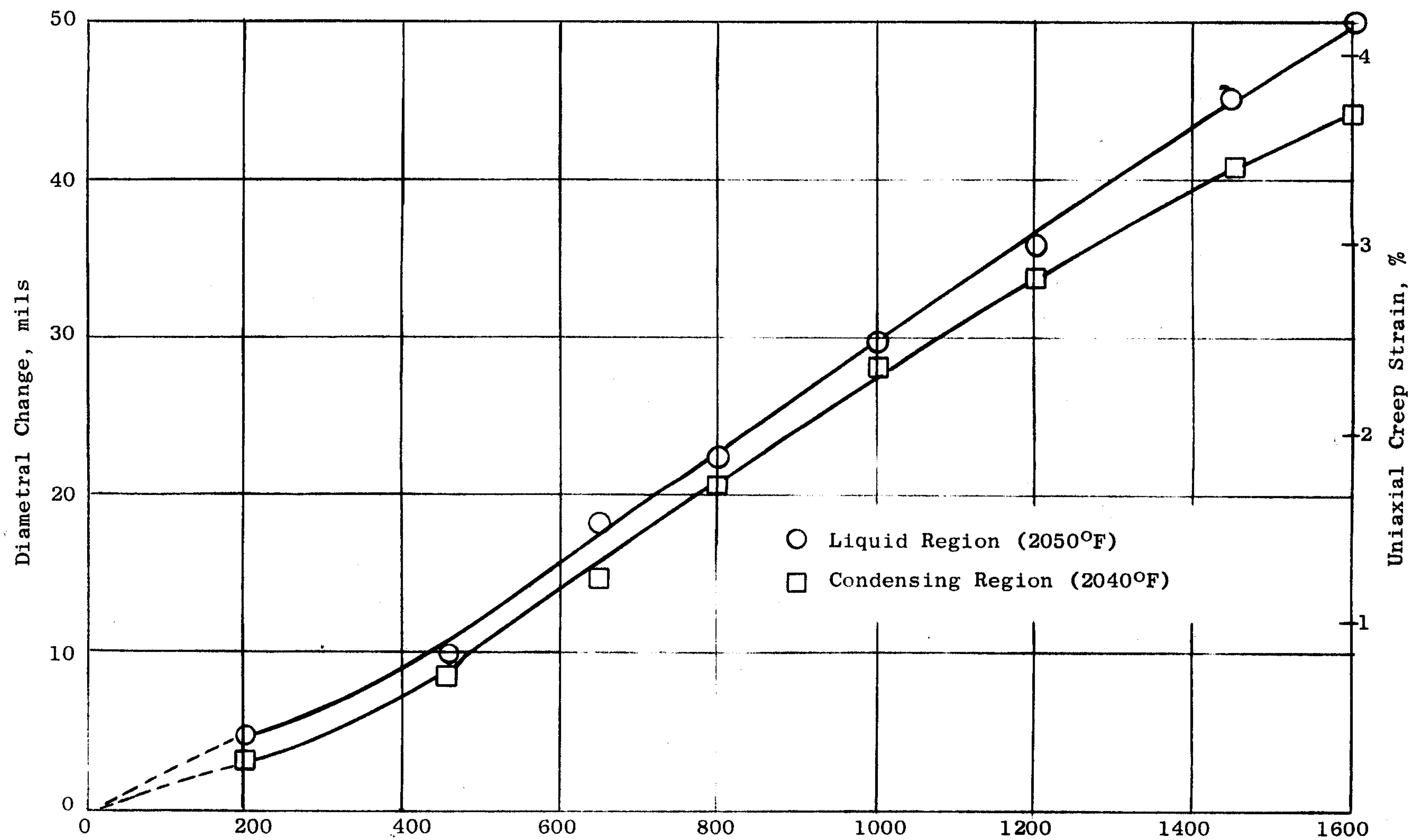

Figure 8. Biaxial Creep Data on the D-43 Alloy Stress Corrosion Reflux Capsule Test II - 1600 Hours Exposure to Potassium. Calculated Uniaxial Stress-5320 psi.

V. FUTURE PLANS

Task I Stress Corrosion Reflux Capsule Program

A. Complete post-test evaluation of Capsule Test I including: metallographic examination, electron microprobe analysis and creep testing of machined specimens.

B. Conclude testing of Capsule Test II.

C. Capsule II will be drained of potassium and cleaned. Post-test evaluation will be completed in the next reporting period.

Task II Bimetallic Isothermal Capsule Program

A. The Topical Report will be submitted to the NASA Technical Manager for approval.

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[1] Work Performed Under NASA Contract NAS 3-2140, "Evaluation of AS-55 Alloy and Other High Strength Columbium Alloys for Alkali Metal Containment," July 25, 1962 to September 15, 1964, General Electric Company, Cincinnati, Ohio.

[2] DiStefano, J. R. and Hoffman, E. E., "Corrosion Mechanisms in Refractory Metal-Alkali Metal Systems," Atomic Energy Review, (1964), Vol. 2, No. 1, p 20.

[3] Harrison, R. W., Studies of Alkali Metal Corrosion on Materials for Advanced Space Power Systems, Quarterly Progress Report No. 4 for Period Ending July 26, 1965, NASA Contract NAS 3-6012.

[4] BNL-823 (S-65), Progress Report of the Nuclear Engineering Department, May 1-August 31, 1963, Brookhaven National Laboratory, p 74.

[5] Hoffman, E. E., Potassium Corrosion Test Loop Development, Quarterly Progress Report #7 for Period Ending April 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54735.

[6] DMIC Memorandum 203, "Recent Information on Long-Time Creep Data for Columbium Alloys," Columbus, Ohio.

[7] Harrison, R. W., Studies of Alkali Metal Corrosion on Materials for Advanced Space Power Systems, Quarterly Progress Report No. 2 for Period Ending December 26, 1964, NASA-CR-54281, NASA Contract NAS 3-6012.

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